Capacitive Resonant Mass Sensor with Frequency Demodulation Detection Based on Resonant Circuit

著者	江刺　正喜
journal or publication title	Applied Physics Letters
volume	88
number	5
page range	053116-1-053116-3
year	2006
URL	http://hdl.handle.net/10097/34836

APPLIED PHYSICS LETTERS **88**, 053116 (2006)

Capacitive resonant mass sensor with frequency demodulation detection based on resonant circuit

Sang-Jin Kim,[a] Takahito Ono, and Masayoshi Esashi
Graduate School of Engineering, Tohoku University, 6-6-01 Aza Aoba, Aramaki, Aoba-ku, Sendai 980-8579, Japan

(Received 21 September 2005; accepted 24 December 2005; published online 1 February 2006)

In this letter, capacitive mass sensing with a 250-nm-thick single-crystalline silicon cantilever is investigated. The mass sensor employs the frequency modulation detection method using an electrical LC oscillator, in which the capacitance of the sensor serves as the component of the oscillator. The displacement noise of the demonstrated capacitive detection is $0.05\ \mathrm{nm}/(\mathrm{Hz})^{0.5}$, which is equivalent to the capacitance change of 2.4×10^{-21} F. It is experimentally shown that the capacitive detection is less affected to temperature fluctuation noise than optical detection. The detectable minimum mass of 1×10^{-14} g is achieved using capacitive detection in ambient atmosphere. © *2006 American Institute of Physics*. [DOI: 10.1063/1.2171650]

Mass sensors based on a micro/nanomechanical resonator have been expected to achieve an extreme performance as required in chemical, environmental, medical fields, etc.,[1–8] which can be achieved by recent developments in a micro/nanofabrication technology. It has been reported that an order of femtograms of mass detection was achieved under ambient pressure and temperature using a bimetal silicon cantilever[5] and an even higher mass resolution of 10^{-18} g was achieved in vacuum.[6,7] These sensors employ tracking the resonance frequency shifts of the resonator in a simple harmonic resonance mode due to mass changes,[9] and have the fundamental issue that the environment has a large influence on the performance of sensor. Especially, in atmospheric pressure, quality factor (Q factor) declines due to air damping[10] and the detectable minimum mass increases. Various noise sources including thermomechanical noise and temperature fluctuation noise limit the resolution of mass detection.[11,12] Miniaturization of the resonator is an effective way to decrease the influence of the thermomechanical noise. However, the temperature fluctuation noise increases as the sensor is miniaturized, especially if its measurement method, such as optical sensing, generates heat and heat conduction.[13] From this point of view, capacitive sensing is less affected to temperature fluctuation noise than other detection methods because it does not employ an optical source. One of the important applications of mass sensors is the characterization of nanomaterials. A hydrogen storage capacity of a carbon nanotube bundle, which was placed at the end of a cantilevered mass sensor, was evaluated.[6] Also a thermal mass desorption spectrum of a pico gram sample was demonstrated by heating a silicon mass sensor;[14] such applications are the driving force to develop our sensitive mass sensors.

In this work, we present and demonstrate the operation of a capacitive mass sensor with a very thin single-crystalline silicon resonator and show that capacitive detection is less affected to noise than optical detection in ambient atmosphere by the comparison of experimental results of both methods. The capacitive mass sensor adopts silicon cantilever as a resonator with capabilities of capacitive readout and electrostatic actuation as shown in Fig. 1. The electrical capacitance between the cantilever and the detection electrode is employed as the component of an electric LC Clapp oscillator with a high resonant frequency,[15] therefore, the resonant frequency of the LC oscillator depends on the capacitance of the sensor. Hence, under the self-oscillation of the sensor, the resonant frequency of the LC oscillator is frequency modulated, and the vibration signal of the cantilever is obtained by demodulating its signal from the LC oscillator. The frequency change of mechanical resonance due to mass loading is measured using a frequency counter. The scanning electron microscope image of fabricated mass sensor is shown in Fig. 2. The details of the fabrication can be found elsewhere,[16] and will be reported at length in the near future.

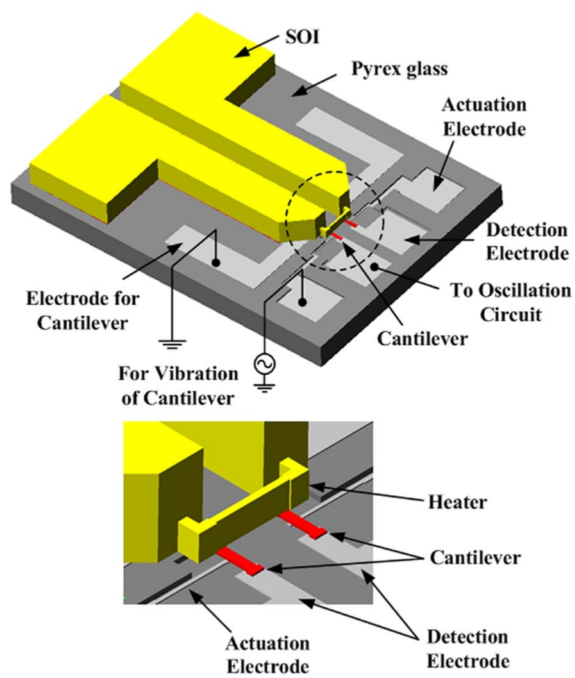

FIG. 1. Schematic figure of the mass sensor consists of two cantilevers for mass detection and reference. The cantilever has two opposite electrodes for capacitive readout and electrostatic actuation, which were fabricated on a Pyrex glass substrate.

[a]Author to whom correspondence should be addressed; electronic mail: sjkim@mems.mech.tohoku.ac.jp

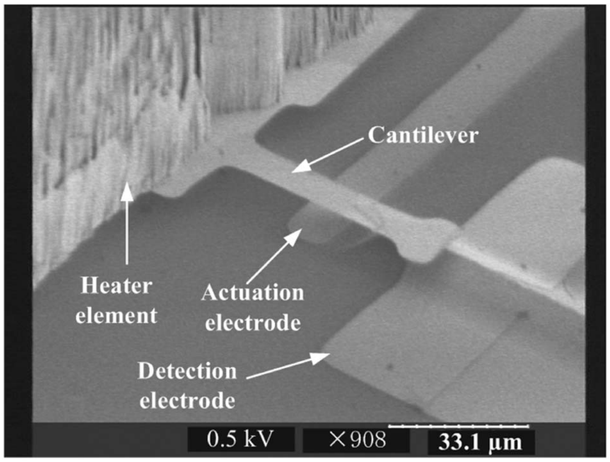

FIG. 2. Magnified scanning electron microscopy image of the fabricated sensor. Starting material is silicon on insulator with a (100)-oriented *p*-type 340-nm-thick top layer, 2-μm-thick intermediate SiO_2 layer, and 300-μm-thick handling layer. The sensing electrode patterned on Pyrex glass substrate opposite to the cantilever with a 7 μm gap distance was 15 μm $\times$ 18 μm in size, and its calculated capacitance is 3.4×10^{-16} F.

The detection of the minimum mass loading in a resonating sensor is limited by frequency noise arising from mainly three noise sources: (1) thermomechanical noise, (2) temperature fluctuation noise, and (3) gas desorption and adsorption noise. The frequency noise due to thermomechanical noise $\Delta f_{\mathrm{noise}}$ is given by[9]

$$\langle(\Delta f_{\mathrm{noise}})^2\rangle = \frac{f_0 k_B T B}{2\pi k Q \langle z_{\mathrm{osc}}^2\rangle}, \tag{1}$$

where k_B is Boltzmann constant, T is the temperature, B is the bandwidth, k is the spring constant of the cantilever, Q is the quality factor, $\langle z_{\mathrm{osc}}^2\rangle$ is the mean-square amplitude of vibration at the end of the beam. Using this Eq. (1) the detectable minimum mass Δm_{min} taken into account the thermomechanical noise is expressed as follows:[14]

$$\Delta m_{\mathrm{min}} = \frac{2G}{(\pi f_0)^{2.5}}\sqrt{\frac{k k_B T B}{\tau Q \langle z_{\mathrm{osc}}^2\rangle}}, \tag{2}$$

where τ is the signal integrating time.

Mass loading Δm is given by the change of the resonance frequency

$$\Delta m = \frac{Gk}{\pi^2}\left(\frac{1}{f'^2} - \frac{1}{f_0^2}\right), \tag{3}$$

where G is the value depending on its geometry of the cantilever. In the case that square beam is employed and mass is uniformly loaded on the cantilever, this factor is given by $G=1.37$. f' is the resonance frequency after mass loading, and f_0 is the resonance frequency before mass loading. Eq. (3) can be approximated as follows:

$$\Delta m = 2\frac{Gk}{\pi^2}\frac{\Delta f}{f_0^3}, \tag{4}$$

where $\Delta f = f - f'$.

As a resonator for mass sensor, the cantilever with a length, width, and thickness of 67, 8 μm, and 250 nm, respectively, was used. The fundamental resonant frequency of this resonator was approximately 78 kHz and the calculated spring constant of the beam is 0.018 N/m. The measured Q factor of the fundamental flexural mode was 10 in ambient pressure. A Clapp oscillator as the electrical oscillation circuit was adopted for capacitive displacement detection, in which the capacitive sensor was employed as the capacitive element of the LC oscillator, as shown in Fig. 3. The resonant frequency from oscillation circuit was approximately 1 GHz. The output signal of LC oscillation circuit is down-converted to 500 kHz by a mixer. The capacitance variation of the sensor, which modulates the resonant frequency of the oscillator circuit; therefore, the vibration signal was detected by a frequency modulation (FM)-demodulator circuit. This vibration signal feeds back to an actuator for actuation via a phase and gain adjuster, which self-oscillates the cantilever near the fundamental resonant frequency of 78 kHz. The oscillation frequency of the sensor was measured using a frequency counter. From a spectrum analysis, the center frequency (ω_0) of the output signal of the LC oscillation circuit shifted to ω_0' due to the change of the average capacitance in vibration. Side band peaks appeared at $\omega_0' - \omega_m$ and $\omega_0' + \omega_m$ because the LC oscillation circuit is modulated in the frequency domain by the vibration of the cantilever. The noise amplitude of the sensor output was evaluated from the spectrum of FM-demodulated signal without the vibration of the cantilever. It was found that the noise amplitude of FM demodulator corresponds to a vibration amplitude of $0.05\ \mathrm{nm/(Hz)^{0.5}}$ in the frequency domain. This vibration noise amplitude is equivalent to the capacitance variation of 2.4×10^{-21} F.

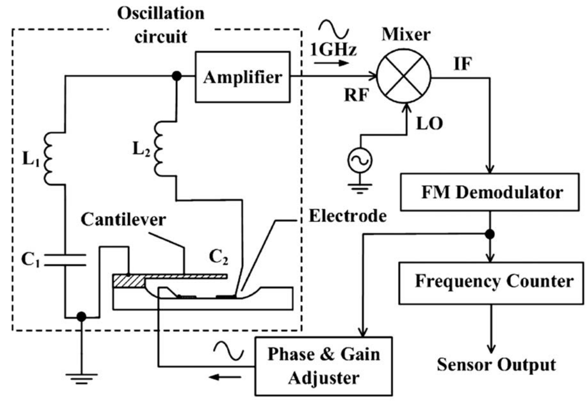

FIG. 3. Measurement system of the sensor. As a LC oscillation circuit, the Clapp oscillator was adopted. FM demodulation was performed by Foster-Seeley discriminator.

The advantage of the capacitive detection technique compared with an optical detection method, is less influence of local heating effect and involved temperature fluctuation noise due to laser irradiation. The fluctuation of heat flow from the cantilever to the support and ambient atmosphere is the origin of the temperature fluctuation noise in a thermal equilibrium. In contrast, the charge fluctuation in the capacitance causes a capacitance noise in capacitive detection method.

The detectable minimum mass using the identical cantilever beam in self-oscillation was investigated on capacitive and optical detections from the frequency noise. In both cases, the vibration amplitude $\langle z_{\mathrm{osc}}\rangle$ was adjusted to 230 nm. The setup for capacitive detection is shown in Fig. 3. Experimental setup of optical detection was described in detail elsewhere.[17] Simply, the detected vibration signal using a laser Doppler vibrometer was fed to a piezoelectric actuator

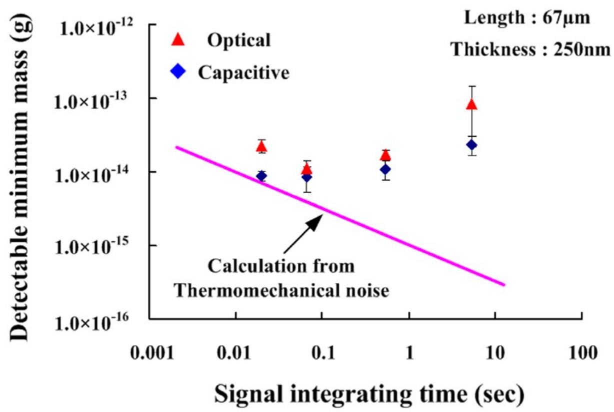

FIG. 4. Detectable minimum mass of the piezoelectrically self-oscillated cantilever measured through both optical detection and capacitive detection using a frequency counter as a function of the signal integrating time.

via the phase and gain adjuster, thus self-oscillation was achieved. The wavelength of the laser (He–Ne) was 633 nm and the power was 0.11 mW, which was focused at the end of the cantilever beam. The frequency noise was measured using the frequency counter as well. Both measurements were performed in ambient atmosphere at room temperature ($\sim$300 K) in a metal chamber. Figure 4 shows the detectable minimum mass measured by both optical and capacitive detections as a function of the time constant of the signal integrating time in frequency detection. The theoretical limit of detectable minimum mass originated in thermomechanical noise was calculated taking into account air damping and squeeze effect from Eq. (2) and plotted in the figure.[18–20] It can be seen that the detectable minimum mass of the capacitive detection was slightly smaller than that of optical detection. The obtained detectable minimum mass of capacitive detection is close to that of calculated result at the short integration time of 0.02–0.08 s. Using the capacitive detection, the detectable minimum mass of below 1×10^{-14} g was obtained with a time constant of 0.08 s. As the signal integration time increase, the difference between experimental and theoretical values increases in both methods. This can be considered that flicker-type frequency fluctuation, which exhibits larger noise at smaller frequency, is caused due to gas adsorption and desorption process on the surface of mass sensor.[21] This frequency fluctuation limits the detectable minimum mass of sensor in actual use in ambient atmosphere. This low frequency noise depends on the adsorption energy of gas molecules on surface, pressure and temperature of environment.[21] In order to identify the dominant noise mechanism exactly, further investigations are necessary.

In conclusion, a single-crystalline silicon resonator as a mass sensor with capabilities of capacitive readout and electrostatic actuation was presented and demonstrated. Furthermore, it was shown that capacitive detection is less affected to noise than optical detection from the comparison of experimental results. In the self-oscillated cantilever with a thickness of 250 nm, the detectable minimum mass of 1×10^{-14} g was obtained using the capacitive detection method. The noise amplitude of the sensor output corresponds to vibration amplitude of 0.05 nm/(Hz)$^{0.5}$ in the frequency domain in comparison with the actuation signal, which is equivalent to the detectable minimum capacitance variation of 2.4×10^{-21} F. These results show the high potential ability of capacitive silicon resonator for high sensitive mass sensor.

Part of this work was performed in the Venture Business Laboratory (VBL) of Tohoku University. This work was supported in part by a Grant-in Aid for Scientific Research from the Japanese Ministry of Education, Culture, Sports, Science and Technology of Japan.

[1]A. Gupta, D. Akin, and R. Bashir, Appl. Phys. Lett. **84**, 1976 (2004).
[2]F. M. Battiston, J. P. Ramseyer, and H. P. Lang, Sens. Actuators B **77**, 122 (2001).
[3]B. Ilic, D. Czaplewski, M. Zalalutdinov, and H. G. Craighead, J. Vac. Sci. Technol. B **19**, 2825 (2001).
[4]H. Jensenius, J. Thaysen, A. A. Rasmussen, L. H. Veje, O. Hansen, and A. Boisen, Appl. Phys. Lett. **76**, 2615 (2000).
[5]N. V. Lavrik and P. G. Datskos, Appl. Phys. Lett. **82**, 2697 (2003).
[6]T. Ono, X. Li, and H. Miyashita, Rev. Sci. Instrum. **74**, 1240 (2003).
[7]K. L. Ekinci, X. M. H. Huang, and M. L. Roukes, Appl. Phys. Lett. **84**, 4469 (2004).
[8]E. Forsen, G. Abadal, S. Ghatnekar-Nilsson, J. Teva, R. Sandberg, F. Perez-Murano, L. Montelius, N. Barniol, and A. Boisen, Appl. Phys. Lett. **87**, 043507 (2005).
[9]T. R. Albrecht, P. Grütter, D. Horne, and D. Rugar, J. Appl. Phys. **69**, 668 (1991).
[10]K. Y. Yasumura, T. D. Stowe, E. M. Chow, and T. Pfafman, J. Microelectromech. Syst. **9**, 117 (2000).
[11]A. N. Cleland and M. L. Roukes, J. Appl. Phys. **92**, 2758 (2002).
[12]K. L. Ekinci, Y. T. Yang, and M. L. Roukes, J. Appl. Phys. **95**, 2682 (2004).
[13]J. R. Vig and Y. Kim, IEEE T. Ultrason. Ferr. **46**, 1558 (1999).
[14]T. Ono and M. Esashi, Meas. Sci. Technol. **15**, 1997 (2004).
[15]S. J. Kim, T. Ono, and M. Esashi, *Transducer'05 The 13th International Conference on Solid-State Sensors, Actuators and Microsystems, 2005*, Vol 1, p 256.
[16]Y. Cho, K. Fujimoto, Y. Hiranaga, Y. Wagatsuma, A. Onoe, T. Terabe, and K. Kitamura, Nanotechnology **14**, 637 (2003).
[17]T. Ono and M. Esashi, Rev. Sci. Instrum. **74**, 5141 (2003).
[18]H. Hosaka and K. Itao, Sens. Actuators, A **A49**, 87 (1995).
[19]F. R. Blom, S. Bouwstra, M. Elwenspoek, and J. H. J. Fluitman, J. Vac. Sci. Technol. B **10**, 19 (1992).
[20]L. D. Landau and E. M. Lifshitz, *Fluid Mechanics, Course of Theoretical Physics* Vol. 6, 2nd ed. (Pergamon, Oxford, 1963).
[21]Y. K. Yong and J. R. Vig, IEEE T. Ultrason. Ferroelect. **37**, 543 (1990).